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3	US 5626650 A	9					USPAT
4	US 5543378 A	7					USPAT
5	US 20030118015 A	9					US-PG
6	US 20030108480 A	9					US-PG
7	US 20030099592 A	9					US-PG
8	US 20020175323 A	30					US-PG
9	US 20020046953 A	17					US-PG

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TITLE: Catalyst-induced growth of carbon nanotubes on tips of cantilevers and nanowires

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REFERENCE-FIGURES: 1

ABSTRACT:

A method is described for catalyst-induced growth of carbon nanotubes, nanofibers, and other nanostructures on the tips of nanowires, cantilevers, conductive micro/nanometer structures, wafers and the like. The method can be used for production of carbon nanotube-anchored cantilevers that can significantly improve the performance of scanning probe microscopy (AFM, EFM etc). The invention can also be used in many other processes of micro end/or nanofabrication with carbon nanotubes/fibers. Key elements of this invention include: (1) Proper selection of a metal catalyst and programmable pulsed electrolytic deposition of the desired specific catalyst precisely at the tip of a substrate, (2) Catalyst-induced growth of carbon nanotubes/fibers at the catalyst-deposited tips, (3) Control of carbon nanotube/fiber growth pattern by manipulation of tip shape and growth conditions, and (4) Automation for mass production.

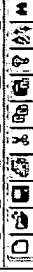
CROSS-REFERENCE TO RELATED APPLICATIONS

1. Invention: Micro. 2. Existing: CA. 3. New case int. 4. (3)4 pro3m. 5. Content Info. 6. Classify: M. 7. EAST: [Data] 8. 09873928.7 9. 09873928.7 10. 09873928.7 11. 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1. Make sure 2002/010554 is carbon nanotubes
2. it looks like non-inventor for US2
3. look for previous CVD d4
4. figure shows for US5
5. look for tip for d6

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1	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20030148577	20030807	18	Controlled alignment of catalytically grown Carbon nanobimorph actuator and sensor	438/238			Merkulov, Vladimir I. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
2	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20020180306	20021205		Carbon nanobimorph actuator and sensor	310/302			Hunt, Brian D. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
3	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20020167375	20021114	17	Carbon nanotube array RF filter	333/186			Hoppe, Daniel J. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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5	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20020117951	20020829		Carbon tips with expanded bases	313/309	313/311		Merkulov, Vladimir I. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
6	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20020046953	20020425		Catalyst-induced growth of carbon nanotubes on tips of	205/104	205/157; 205/192;		Lee, James Weifu et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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8	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6649431 B2	20031118		Carbon tips with expanded bases grown with	438/20	257/13; 257/79;		Merkulov, Vladimir I. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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2	US 20030148577 A	18	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>			US-PG
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7	US 20020046953 A	17	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>			US-PG

[0024] The method can include two main steps: formation of a precisely placed single metal catalyst dot on a substrate by utilizing EBL in conjunction with electron-gun metal evaporation; and catalytic growth of a NCNT on the substrate using PE-CVD.

[0025] Growth of CNTs and CNFs requires the presence of a catalytic precursor. Ni can be used as a catalyst. However, other metals such as Fe, Co, etc. can also be utilized as the catalyst with these procedures, the results being similar.

[0026] In order to grow a single carbon nanotube (CNT) or CNF, formation of a single catalyst nanoparticle (or cluster) may be required. This catalyst nanoparticle is formed by forming a metal catalyst dot with width (diameter) D, and film thickness, T. FIGS. 1A-1P show fabrication of vertically aligned carbon nanofibers (VACNFs) utilizing plasma enhanced chemical vapor deposition (PECVD). 1E-1P preceded by electron-beam lithography and metal evaporation (FIGS. 1A-1D). A catalyst dot 150 is fabricated on a substrate using electron beam (e-beam) lithography and electron gun (e-gun) metal evaporation as shown in FIGS. 1A-1D. A substrate 110 is first coated with an e-beam resist 120 (e.g. PMMA); the resist 120 is then e-beam exposed and developed (FIGS. 1A and 1B), to produce a small opening 130 in the resist 120 with width (diameter) D. A buffer layer 140 (Ti in this case) is deposited next to prevent the formation of catalyst silicide and to impede catalyst diffusion at elevated temperatures. Next, a catalyst layer 150 (Ni, in this case) is deposited (FIG. 1C). Finally, a single, isolated catalyst dot is obtained by lifting off the metal-coated resist in acetone (FIG. 1D). Multiple dots, or pattern arrays of dots, also can be produced by this process.

[0027] FIGS. 1E-1P show a VACNF is prepared by PECVD in a vacuum chamber 160. The vacuum chamber 160 can include an anode 162 and a cathode 164. The cathode 164 can also function as a heater. One of the advantages of this method is that high vacuum is not required, i.e. the chamber 160 can be evacuated using only a mechanical pump (FIGS. 1E and 1P). Upon ammonia plasma pre-etching (alternatively, hydrogen and other gases can be used) and annealing the Ni/Ti assembly on Si at the elevated temperatures required to grow VACNFs (about 700 degree C. in this case), the Ti layer 140 continues to adhere to the Si substrate 110, whereas the initially continuous Ni layer 150 breaks into one or more little nanoparticle droplets (FIG. 1E).

[0028] FIGS. 2A-2D show scanning electron microscopy images of single and multiple vertically aligned carbon nanofibers formed from a single or multiple catalyst dot. Upon heating and ammonia plasma pre-etching the catalyst layer breaks into nanodroplets. Each nanodroplet provides for catalytic growth of an individual nanofiber. This droplet is the necessary precursor for the catalytic growth of a single VACNF at this predetermined location. Within an initially large dot, multiple droplets are formed (FIG. 2A). See FIG. 2B also. However, below a critical dot size only a single nanoscale Ni droplet forms (FIG. 2B) and consequently only a single nanofiber is grown. See FIG. 2D also. The critical dot size, and the size of its resulting Ni droplet, will depend upon the choice of the buffer layer between the catalyst and the substrate, the type and thickness of the catalyst used, and the annealing/growth temperature. For example, for growth of a single VACNF at 700 degree C. using an initially 15 nm thick Ni catalyst on a Ti buffer layer on Si, the critical dot size (diameter) is about 350 nm. The diameter of the Ni nanoparticle droplet formed is about a factor of 3 smaller, about 100 nm in this example. Smaller catalyst nanoparticles can be obtained by initially forming a smaller catalyst dot. For instance, 100 nm dots with a 10 nm thick Ni layer produce Ni droplet

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Merkulov et al.

(54) CARBON TIPS WITH EXPANDED BASES

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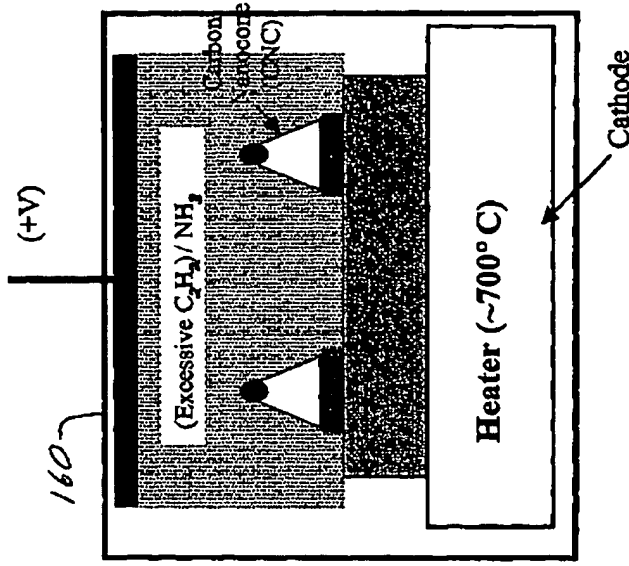
(57) ABSTRACT

Systems and methods are described for carbon tips with expanded bases. A method includes producing an expanded based carbon containing tip including: fabricating a carbon containing expanded base on a substrate; and then fabricating a carbon containing fiber on the expanded base. An apparatus includes a carbon containing expanded base coupled to a substrate; and a carbon containing fiber coupled to said carbon containing expanded base.

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[0032] The invention can include catalytically-controlled growth of vertically aligned (i.e. substantially perpendicular to the substrate surface), multi-walled CNRs (Merkulov et al., 2000). Referring to FIG. 2, a PE-CVD process for growing MWNT in place in a prefabricated electrode structure is depicted. A vacuum chamber 200 includes an anode 210 and a cathode 220. A substrate 230 is located on the cathode 220. A barrier layer 235 is formed on the substrate 230. A plurality of dielectric layers 240 alternating with a plurality of extractor layers 245 compose an electrode structure 250. The process utilizes plasma-enhanced chemical vapor deposition (PE-CVD) in conjunction with evaporation and precise placement of a metal-catalyst dot 255. The catalyst dot 255 can be a nickel catalyst particle that is determined by lithographic definition. A mixture of acetylene (C₂H₂, sub. 2) and ammonia (NH₃, sub. 3) is used as a source gas during the PE-CVD process. The substrate 230 is heated above about 600 degrees C. After the plasma is turned on, MWNTs grow selectively at the locations where catalyst dots (e.g., Ni, Fe, Co) were pre-deposited. A single self-aligned MWNT 260 can be grown in place (in situ) via the catalyst dot 255. The MWNT 260 can be carbon containing or substantially pure carbon. The method depicted in FIG. 2 can also be used to fabricate self-aligned single wall carbon containing nanotube and/or self-aligned carbon containing nanofibers. Similarly, these MWNTs and/or NFs can be carbon containing or substantially pure carbon.

[0033] In the method depicted in FIG. 2, no hot filament is present and the substrate 230 is heated directly by placing them on a heater plate (e.g., the cathode 220). This technique has the advantages of growth-temperature control and large-area deposition (not possible with a hot filament) and therefore creates the possibility of industrial-scalable device fabrication.

[0034] FIGS. 3A-3G show a fabrication process for a MWNT-based vacuum nanoelectronic device. The device depicted in FIGS. 3A-3G is a simple gated electron emitter structure that can be the basis for more complex vacuum nanoelectronic devices. In FIGS. 3A-3P, an electrode structure is defined through lithographic steps. In FIG. 3A, a buffer layer 305 is deposited on a substrate 300. The substrate 300 can be 4" n-Si and the buffer layer 305 can be a titanium-tungsten alloy. In FIG. 3B, alignment mark 310 are formed on the buffer layer 305. The alignment marks 310 can include gold and could be conductive traces. The alignment marks are for subsequent photolithography and critical dimension (CD) measurements. Referring to FIG. 3C, one of the steps in this process is the definition of a catalyst particle 320. The catalyst particle can include one, or more, of nickel, iron and cobalt. The catalyst can be a round dot of approximately 100 nm diameter on a 50 micron pitch. The catalyst particle can be one member of an array where the dots are arranged on a pitch of approximately 50 microns. FIG. 3D shows a dielectric layer 330 deposited over the catalyst particle 320, the traces 310 and the buffer layer 305. The dielectric layer can include silica. In this case, the silica can be deposited using PECVD at a temperature of approximately 275 degrees C. FIG. 3E shows an extractor layer 340 deposited over the dielectric layer 330. The extractor layer 340 can be a chromium layer approximately 150 nm thick. There is an extractor aperture 345 formed in the extractor layer 340. The extractor aperture 345 can be formed using a spin-on photoresist. FIG. 3F shows a dielectric well 350 formed in the dielectric layer 330. The dielectric well can be formed using reactive ion etching using the extractor layer 340 as an etch mask. The formation of the dielectric well should uncover the catalyst particle 320 to catalyze growth. The dielectric well 350 can be coincident with the extractor aperture 345. Referring to FIG. 3G, after the entire electrode structure is fabricated, and the catalyst is uncovered, a MWNT 360 is grown in place (in situ) within the electrode structure. The growth can be by PECVD at a

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FIELD EMISSION DEVICES HAVING CARBON CONTAINING TIPS

(57) ABSTRACT

Systems and methods are described for field emission devices having carbon nanotube emitters. A method, includes: providing a substrate; depositing a catalyst, the catalyst coupled to the substrate; depositing a dielectric layer, the dielectric layer coupled to the substrate; depositing an extractor layer, the extractor layer coupled to the dielectric layer; forming an extractor aperture in the extractor layer; forming a dielectric well in the dielectric layer to uncover at least a portion of the catalyst; and then fabricating a carbon containing tip (i) having a base located substantially at the bottom of the dielectric well and (ii) extending substantially away from the substrate. An apparatus, includes: a substrate; an electrode structure coupled to the substrate, the electrode structure including a dielectric layer coupled to the substrate, the dielectric layer including a dielectric well that is formed in the dielectric layer after the dielectric layer is deposited; and an extractor layer coupled to the dielectric layer, the extractor layer including an extractor aperture; and a carbon containing tip coupled to the substrate, the carbon containing tip having a base located substantially at a bottom of the dielectric well and extending substantially away from the substrate, the carbon containing tip being grown from the bottom of the dielectric well using a catalyst that is introduced at the bottom of the dielectric well after the dielectric well is formed. A method, includes: providing a substrate on a heater plate in a vacuum chamber; providing a carbon source gas and an etchant gas; heating the substrate with the source gas and the etchant gas; heating the substrate with the source gas and the etchant gas; and then fabricating a carbon tip on the substrate with the carbon source gas and the etchant gas using plasma enhanced chemical vapor.

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(21) Appl. No.: 09/810,531

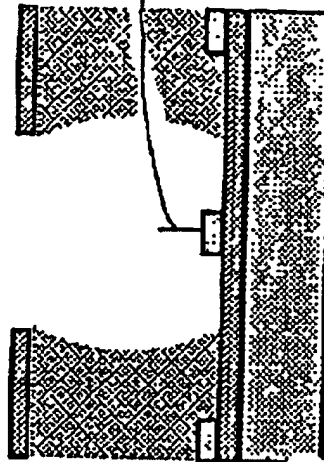
(22) Filed: Mar. 15, 2001

Related U.S. Application Data

(63) Non-provisional of provisional application No. 60/228,713, filed on Aug. 29, 2000.

Publication Classification

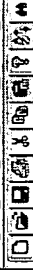
(51) Int. Cl.⁷ H01J 1/02
(52) U.S. Cl. 313/309



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U	11	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current	XRef	Retrieval	C	Inventor	S	C	
1				US 20020127725 A1	20020912	14	Process for testing catalysts using thermography	436/37	422/104;				Willson, Richard C.			
2				US 20020014413 A1	20020207		Electrolytic system and methods for screening	205/81	204/229.8				Symons, Peter G. et al.			
3				US 6630111 B1	20031007		Apparatus for testing catalysts using spectroscopy	422/130	422/129;	422/196;			Willson, III, Richard C.			
4				US 6627571 B1	20030930		Method and system for the situ synthesis of a	502/2	502/180;	502/232;			Lugmair, Claus G. et al.			
5				US 6623970 B1	20030923		Process for testing catalysts using spectroscopy	436/37	422/52;	422/62;			Willson, III, Richard C.			
6				US 6623969 B1	20030923		Process for testing catalysts using mass	436/37	250/281;	250/282;			Willson, III, Richard C.			
7				US 6623968 B1	20030923		Parallel flow reactor and apparatus for testing	436/37	250/281;	250/282;			Willson, III, Richard C.			
8				US 6623967 B1	20030923		Process for testing catalysts using	436/37	250/288;	422/69;			Willson, III, Richard C.			
9				US 6605470 B1	20030812		Process for testing catalysts using detection	436/37	436/141;	436/159;			Willson, III, Richard C.			
10				US 6514764 B1	20030204		Catalyst testing process with in situ synthesis	436/37	422/62;	436/147;			Willson, III, Richard C.			
11				US 6333196 B1	20011225		Catalyst testing process and apparatus	436/37	422/104;	422/62;			Willson, III, Richard C.			
12				US 6063633 A	20000516		Catalyst testing process and apparatus	436/37	422/104;	422/62;			Willson, III, Richard C.			

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